GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF CONTRACT ADMINISTRATION
SPONSORED PROJECT INITIATION

Date: May 1, 1979

Project Title: Exploratory Activation of Coal Char

Project No: A-2364

Project Director: S. B. Smith

Sponsor: TIGG Corporation

Agreement Period: From 4/10/79 Until open

Type Agreement: Ltr. dtd. 4/10/79

Amount: $750

Reports Required: Final Letter Report

Sponsor Contact Person (s):

Technical Matters	Contractual Matters (thru OCA)
Donald Tiggelbeck President TIGG Corporation Box 11661 Pittsburg, PA 15228 (412)563-4550	

Defense Priority Rating:

Assigned to: Technology & Development (School/Laboratory)

COPIES TO:

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- Division Chief (EES)
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CA-3 (3/76)

GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF CONTRACT ADMINISTRATION

SPONSORED PROJECT TERMINATION

Date: May 4, 1981

Project Title: Exploratory Activation of Coal Char

Project No: A-2364

Project Director: S. B. Smith

Sponsor: TIGG Corporation

Effective Termination Date: 6/30/80

Clearance of Accounting Charges: 6/30/80

Grant/Contract Closeout Actions Remaining:

NONE

_ Final Invoice and Closing Documents
_ Final Fiscal Report
_ Final Report of Inventions
_ Govt. Property Inventory & Related Certificate
_ Classified Material Certificate
_ Other _____

Assigned to: EMSL/ESG (School/Laboratory)

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TIGG CORPORATION

To: Alice/ARC

Interim Letter Report on Project No. A-2364

"Exploratory Activation of Coal Char" - Steps 1 & 2

By

Dr. S. B. Smith
Robin B. Brown

Evaluation of Results

This 12 x 40 mesh carbon or char appears to be an excellent material for steam activation based on the data thus far determined. The initial ash level is low as well as the Volatile Matter. Though sulfur was not determined, there appeared to be little odor or other evidence of its presence during heating and activation.

Activity Development

This char responds very well to activation with respect to yield for all of the usual criteria; Iodine No, Phenol Capacity or Molasses Decolorizing Index. It is unusual for a char to respond well to all three. It does tend, however, at higher degrees of burnoff to favor DI development over Iodine No., the latter values tending to level out near 1000 mg/g. It is quite surprising that the fine pore development is also good as shown by the Phenol Capacity reaching a maximum as high as 6.6, which is well above "specs" for a premium water carbon (PC=5.0 or MPV=20). By the new AWWA procedure the maximum reached is 1.73 g/l (required to remove 90% of phenol from a 200 mg/l solution) against an AWWA standard of 3.5 g/l.

The Decolorizing Index is reported since our attempts at Molasses Number determination were not acceptable. Though we have the Calgon molasses and Standard Carbon, the directions for molasses preparation give us too dark a color for accurate measurement. (We should be able to correct this in the next phase). With this material, levels of decolorizing activity equivalent to granular coal carbons are easily reached (WVG=8DI units). In fact, the DI levels found in powdered decolorizing carbons can be attained by steam alone at yields of 40 to 50%. These yields should be improved, probably at the expense of IN and Phenol Capacity, by using higher temperatures and more oxidizing atmospheres as suggested for Steps 3 & 4 of the proposal.

Physical Properties

Screen analyses were run on the feed and 45 min. activation product indicating a considerable shrinkage which helps maintain a good Apparent Density. "Log probability" plots of the screen analyses are shown in Figure 3 and the Median Particle Diameters show a shrinkage of 18.8% from 0.80 mm to 0.65 mm. The char, is really too fine to directly produce a good 12 x 40 mesh product. At this average yield of 56.5% a roughly 14 x 50 product results. The density of 0.497 g/cc is quite high for such a material.

The Abrasion No. was rather low for a coal-base carbon at 67%. The carbon has softened considerably in the activation and the Abrasion No is probably higher than warranted because of the bias created by a very fine material in this test. As a granular product it could be used only on a throw-away basis. However, it could be ground to good powdered product.

Recommendations

Since this char responds so well to activation by steam, we suggest the following:

1. Activate at higher temperatures, and with air added, to enhance DI development and consider the product as a powdered decolorizing carbon.

2. From a coarser feed stock (if available) prepare a low activity 12 x 40 granular carbon to be used on a throw-away basis for several years as a topping in sand filters to control tastes and odors in municipal water plants.

3. Proceed with Steps 3-8 in the proposal, but add granular water carbon as a second target for evaluation.

Stanton B. Smith, Ph.D.
Principal Research Scientist

SBS:gp

LABORATORY ACTIVATION OF COAL CHAR at 1700 F

2" Rotary Furnace, Steam only, 2 g/min, 50g charge db

PROPERTY	ACTIVATION TIME				
	0	10	30	45 (6)	60
Yield	100	90.8	68.0	47.8	37.6
Apparent Density, g/cc	.741	.693 (1)	.563 (1)	.497 (2)	.468 (1)
Iodine No., mg/g	342	492	785	983	1008
MPV, (3) ppm	-	23.7	17.6	15.2	16.5
Phenol Cap. 100/MPV	-	4.18	5.70	6.58	6.08
Decolorizing Index, (4) DI units	-	3.0	5.0	12.5	14.0
Volatile Matter, %	5.25*				
Ash, %	2.09*				
Abrasion No., (5) %				67.2 (2)	
Mean Particle Diameter, mm				0.71 (2)(5)	
Moisture, % wb	1.43*				

*Average of 2 determinations

(1) Run on less than 100 ml of sample

(2) Run on combined 4 runs, see(5)

(3) Modified Phenol Value by Westvaco method using 790 factor

(4) Spec. for WVL is 7.0 DI, WVG is 8.0

(5) Four 45 minute runs were pooled to get 100g required
The average yield on these was 56.5

(6) Analyses reported on first run, except where otherwise noted

FIGURE 1

LABORATORY ACTIVATION OF COAL CHAR (Sample #1) at 1700 F

2" Rotary Furnace; Steam only, 2 g/min; 50g charge db

ADSORPTION PARAMETERS VS YIELD

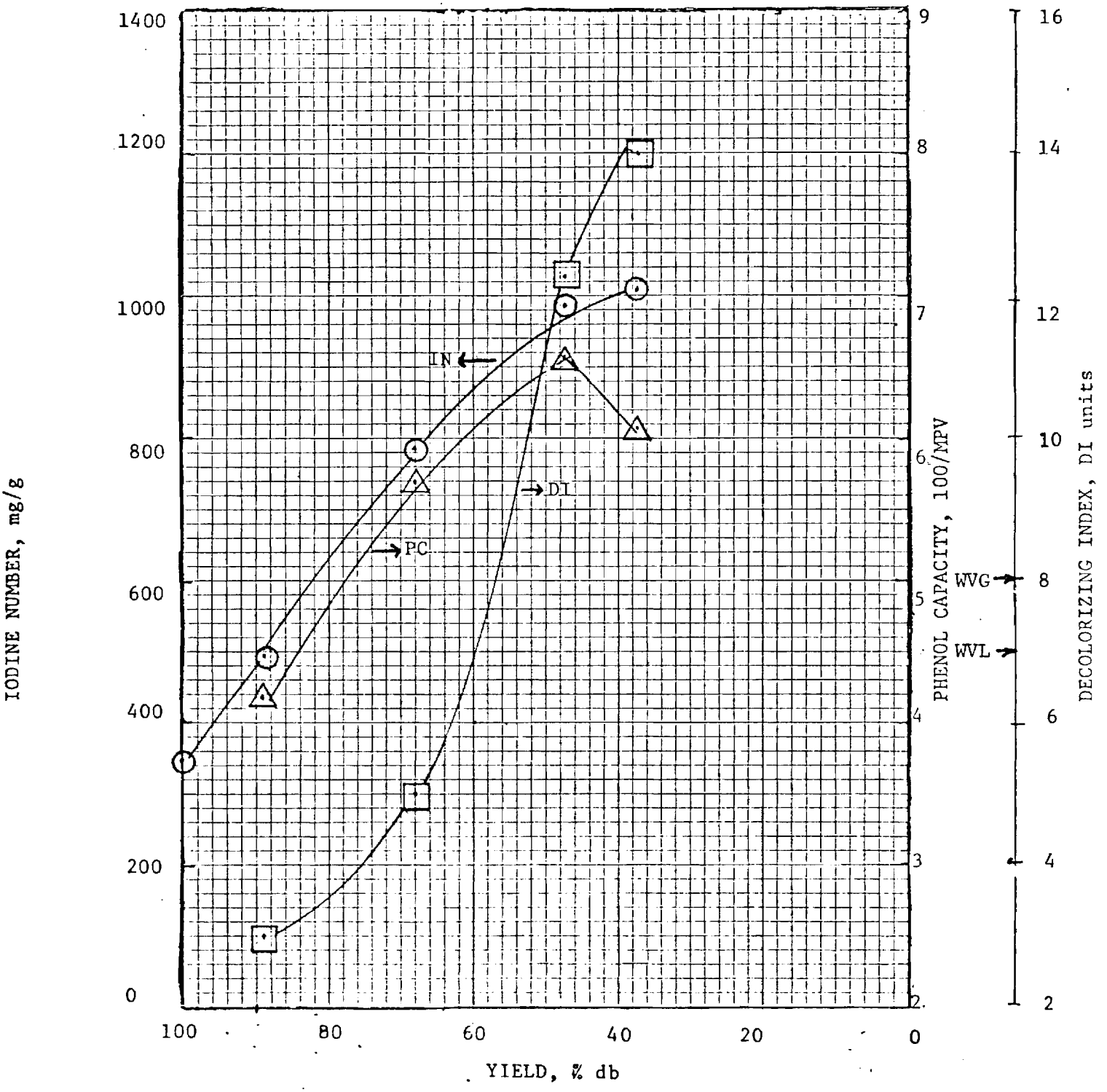
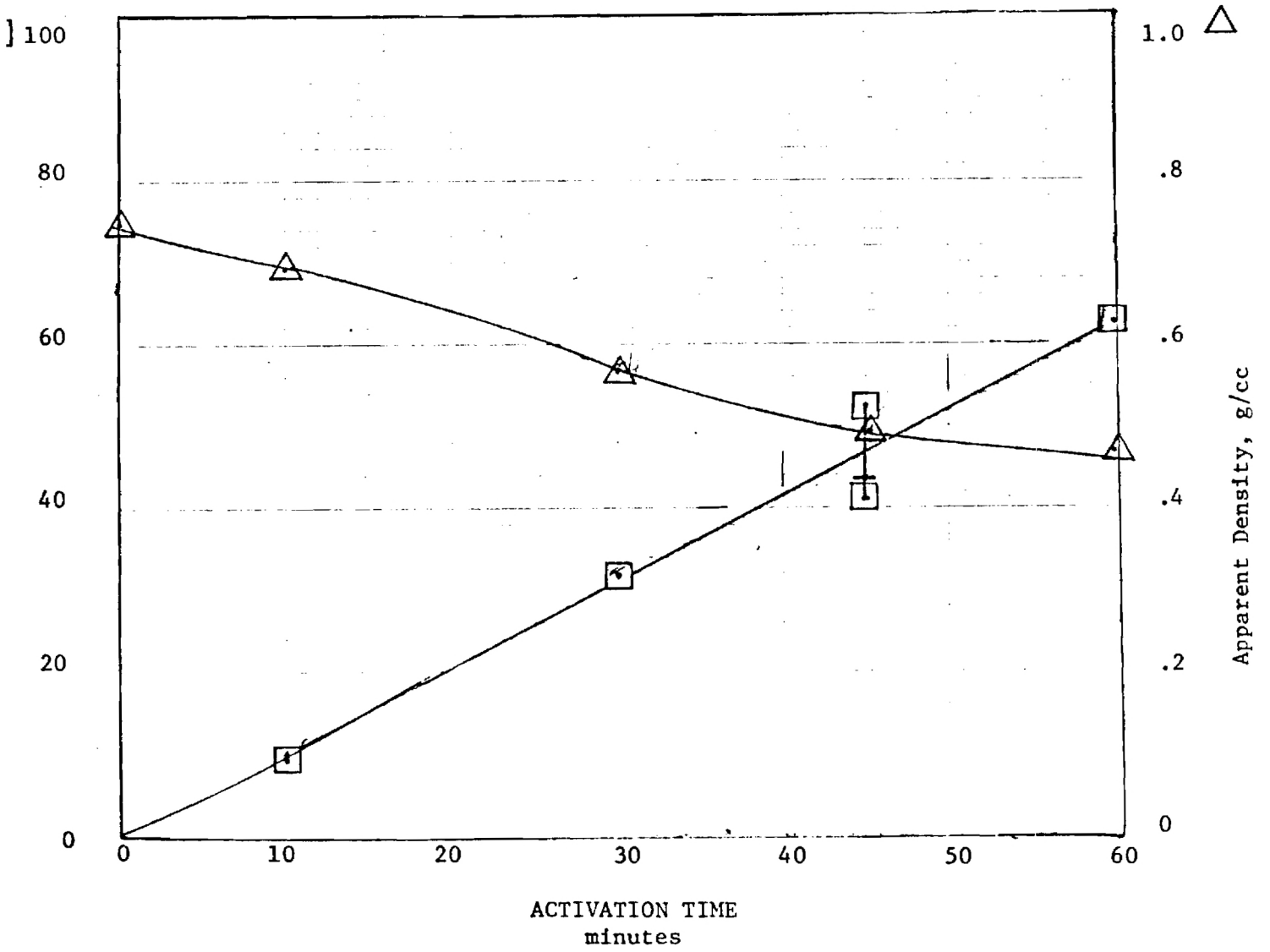


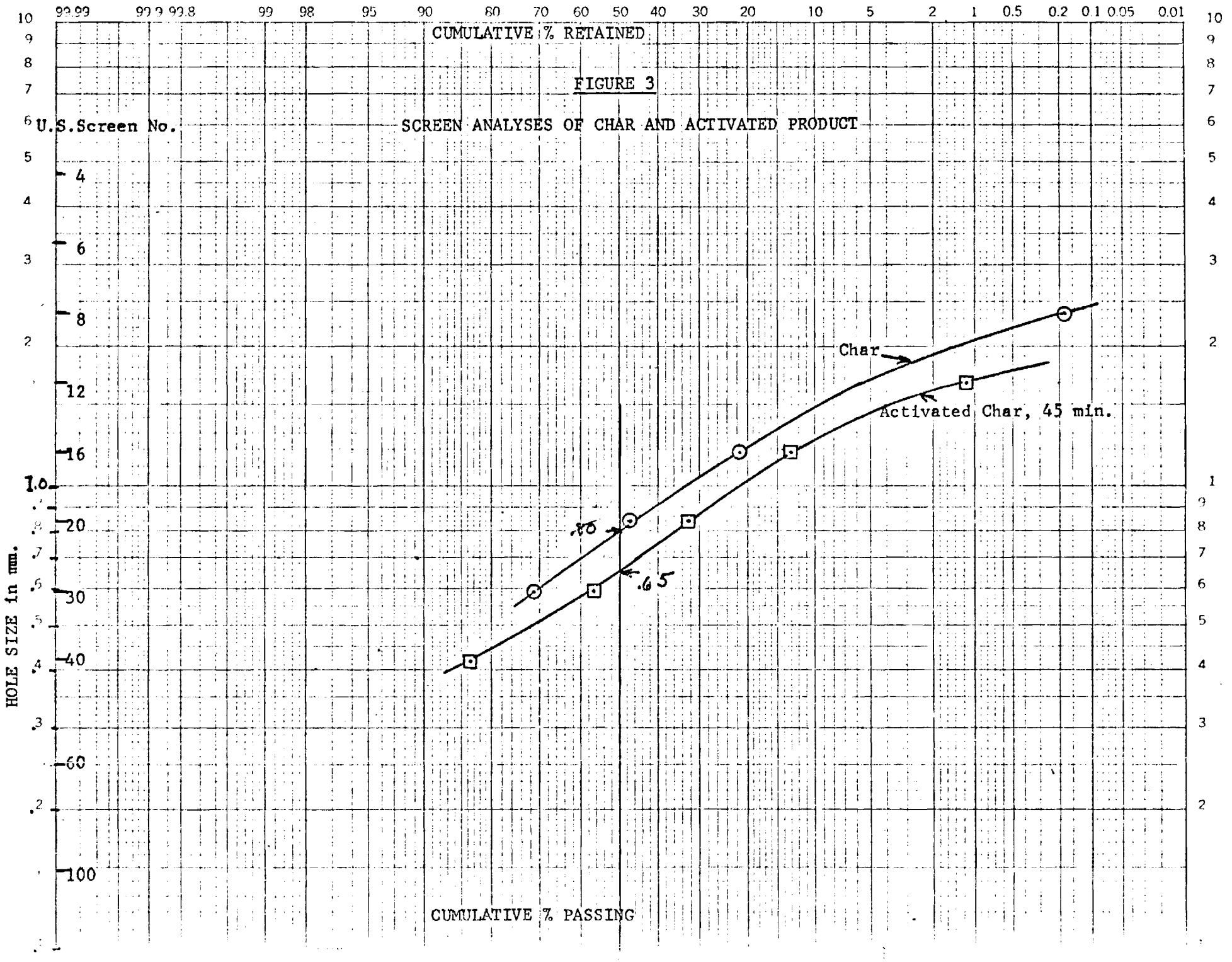
FIGURE 2

BURNOFF VS ACTIVATION TIME

AND

APPARENT DENSITY VS ACTIVATION TIME




CUMULATIVE % RETAINED
FIGURE 3
U.S.Screen No.
SCREEN ANALYSES OF CHAR AND ACTIVATED PRODUCT
99.99 99.9 99.8 99 98 95 90 80 70 60 50 40 30 20 10 5 2 1 0.5 0.2 0.1 0.05 0.01
4
6
8
12
16
20
30
40
60
100
Char
Activated Char, 45 min.
.80
.65
HOLE SIZE in mm.
CUMULATIVE % PASSING

G.R. Harry-C
no encl
A-2364

ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

April 18, 1980

Dr. F. K. McGinnis, Executive Vice President
Shirco, Inc.
2451 Stemmons Freeway
Dallas, TX 15207

Dear Mac:

The attached data should adequately cover the evaluation of the "second regeneration" samples. I will be interested in your observations on the actual run. Hopefully my findings will tie in well with what actually took place.

As you requested, we included in the BET structure tests a calcined "spent" sample. We heated a 50 gram portion in a stream of nitrogen from room temperature to 810°C. Since the furnace heats slowly this procedure took over an hour. A yield of 80.8% was obtained but some of the loss was due to loss of fines carried out by the nitrogen stream rather than to devolatilization of the sample. The structure curves fell well into line but really this did not tell us much in proportion to the added cost. Please give me your comments on this when you call in regard to the next regeneration.

Hopefully, in the next cycle more organic material will be adsorbed. Low pick-up tends to cause some over-regeneration in my opinion. Could the carbon have been left on stream longer in this cycle or was it showing signs of break through?

I was pleased in this set to see the Abrasion No.'s around 50% which is where this type of carbon should fall. It appears that after the wearing and washing away some of the softer material in the 12 x 16 range the remaining carbon is more abrasion resistant.

In regard to the reference sample for Apparent Density determination we obtained on two trials the same value, 0.429g/cc after drying at 105°C for three hours and cooling in a desiccator one hour. This agreed exactly with the virgin sample of this set.

I trust this information is what you need. Do not hesitate to call if there are further questions. Please next time, however, be sure that at least 1/2 pound (dry basis) of each sample is sent. This time we were held up for the second portion which, after drying, was blended with carbon from the first shipment.

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With this reporting completed we will be sending an invoice amounting to $1300.

Thank you for continuing with this program.

Very truly yours,

Stanton B. Smith, Ph. D.
Principal Research Scientist

SBS/pr

ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

April 18, 1980

REPORT TO SHIRCO, INC.

EVALUATION OF TWICE REGENERATED CARBON, HD1030

FROM EVANSVILLE, INDIANA

Project No.: A-2364

General Comments

The results of physical and chemical tests on the regenerated carbon, the spent carbon, laboratory calcined spent carbon and the "new" or "current virgin" carbon indicate that an efficient regeneration was accomplished following the second adsorption cycle. Apparently very little organic matter was adsorbed and as a result a small amount of skeletal carbon was burned, slightly lowering the density. However, on an equal volume basis the surface areas, iodine numbers and molasses decolorizing indeces indicate regeneration efficiencies above the "current virgin" carbon level. The finest pores have been retained as shown by low MPV values. There were no significant changes in the particle diameter and ash content. The pick-up in volatile matter in the spent carbon above the once-regenerated sample from the previous set indicates the expected adsorption, though it is lower than anticipated. There was no significant change in hardness as indicated by the Abrasion Numbers. The screen analysis data show no generation or accumulation of fines.

The pore-size distributions by N_2 desorption are almost identical to the first cycle set. The regeneration appears to open up pores in the 70 to 120Å diameter range and from 30Å on down. As shown in Figure 1 there is no change in porevolume where the two curves remain equidistant (in the verticl direction). Where they diverge there has been an increase in pore volume in this corresponding diameter range. Interestingly, the calcined "spent" sample is almost identical with the "current virgin" sample at the high end but from 45Å down there is a movement away from the "virgin" curve toward the "regenerated" curve. The calcined sample appears to have an uniquely high volume of pores between 40 and 50Å.

In summary it appears that the second regeneration was carried out very

successfully but that the conditions may have been a little too severe in view of the small amount of organic matter accumulated in the adsorption cycle.

TABLE 1

SHIRCO INC., SECOND REGENERATION OF HD1030 ACTIVATED CARBON

SUMMARY TABLE

PROPERTY	Sample:	VIRGIN by weight (by vol.)*	SPENT by weight (by vol.)	REGENERATED by weight (by vol.)
BET Surface Area	m^2/g	516	636 (after cal.)	644
	m^2/ml	(219)	(285)	(260)
Iodine No.	mg/g	439	432	596
	mg/ml	(186)	(187)	(240)
Modified Phenol Value	ppm	51.9	37.2	28.5
Westvaco Meth.)	$ml/10^6g$**	(122.4)**	(85.7)**	(70.7)
WWA	mg/l	5.91	4.24	3.25
Glasses Decolorizing	DI Units	10.2	9.8	14.7
Index		(4.33)	(4.25)	(5.92)
Moisture w.b.	%	53.9	52.6	57.0
	g/100ml	(22.9)	(22.8)	(23.0)
Ash	%	14.8	14.8	15.7
	g/100ml	(6.28)	(6.42)	(6.33)
Volatile Matter	%	11.8	12.4	6.6
	g/100ml	(5.00)	(5.38)	(2.66)
Apparent Density	g/ml	.424	.434 (.448 after calcining)	.403

Screen Analysis

		Virgin	Spent	Regenerated
U.S. Mesh 0 n 10	%	0.1	0	.1
10 x 12	%	2.8	2.8	1.9
12 x 16	%	45.0	37.1	37.8
16 x 20	%	38.9	40.3	43.0
20 x 30	%	10.9	14.3	13.1
30 x 40	%	1.6	3.2	2.5
Through 40	%	0.6	2.2	1.6
Particle Diameter		99.9	99.9	100.0
(Calculated) Mean	mm	1.18	1.11	1.12
(From graph) Median	mm	1.18	1.09	1.10
Erasion Number	% by mean pd	43.6	48.4	50.7
	% by median pd	46.6	52.3	53.6

Values in ( ) calculated by multiplying by the App. Density
Values in ( ) calculated by dividing by the App. Density

Detailed Evaluation (refer to Table 1 for numerical data)

BET Surface Areas measured on the three samples indicate a considerable increase (24.8%) in specific surface of the regenerated sample over that of the virgin carbon. However, due to reduction in density the increase is only 18.7% on a volume basis. The calcined "spent" sample interestingly shows a specific surface very close to the regenerated sample indicating that the accumulated organic matter is quite effectively removed by heat alone. Surprisingly the area per unit volume is higher than the regenerated carbon but this is due to considerable shrinkage which took place during the long heat up period during the calcination.

Perhaps the most meaningful comparison is to be made between this twice-regenerated sample and the product of first regeneration reported in Dec. '79. There has been a decrease on both a weight and volume basis but the twice-regenerated carbon is still above both of the virgin samples in surface area.

Iodine Numbers tell very nearly the same story as the total surface areas, the second regeneration has increased the iodine number over the current virgin carbon but it is not quite up to the standard of the first regeneration or the first virgin sample however, this latter sample was judged to be atypical. On the "spent" sample (not calcined in this case) there is considerable reduction in IN from the first regenerated, though it is not much different from the "current virgin" sample. As is usually the case with large-pored carbons, the BET areas are higher than the iodine numbers.

Modified Phenol Values show a slight impairment of the "spent" sample compared with the first regenerated as expected. The second regeneration has brought the carbon back again to this level, which is considerably better than the "current virgin" sample, but about the same as the "first virgin" sample. This shows that the finest, most retentive pores, are not being lost in the saturation and regeneration process. Again, it appears that there is very little saturation or deactivation of the fine pores in the adsorption cycle.

Molasses Decolorizing Index figures indicate that while the second regeneration has been effective it was not as efficient as the first. The first virgin sample showed a very high DI as compared with the "current virgin" sample. If the latter is more typical, then the overall improvement appears good.

Moisture Content. The water held in the pores of the wetted and drained carbons is almost identical for all on an equal-volume basis.

Ash. The total ash is very nearly constant across both regenerations on an equal volume basis.

Volatile Matter data indicates that the extent of saturation was much less in the second adsorption cycle than the first as already pointed out above.

Apparent Density figures indicate again very little, if any, measureable weight pick up in the second adsorption cycle. As a result there was some burning of skeletal carbon in the second regeneration resulting in a slightly lower final density. Hopefully in the next adsorption cycle a higher degree of saturation will be realized before regeneration. It is interesting that the calcined "spent" sample shows an increase in density even as it was devolatilized due to shrinkage on heating.

Abrasion Numbers. These values were determined on the standard weight of 100g rather than 50g as in the first regeneration set. The values in the 50% range are reasonable for this type of carbon and indicate there has been no softening of the carbon. The values are slightly higher when "median" particle diameters are used since one does not assume the minus 40 mesh fraction to have a diameter of zero as in the calcutation of "mean" diameters. The apparent increase in hardness on regeneration may be due to the density decrease as the abrasion test is density sensitive.

ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

CARBON ANALYSIS REPORT

April 11, 1980

SAMPLE:

Source: Shirco, Inc. Grade (if known): DARCO HD1030

Designation: Evansville, Ind. Virgin HD1030

Project No.: A-2467 Date Rec'd: 2/20/80 and 3/3/80 (Combined)

ANALYTICAL RESULTS (by Westvaco Standard Methods unless otherwise noted)

TEST	Units	RESULTS Replicates (1)	(2)	Average
Abrasion No. (Ro-Tap)	%	43.6		
*Iodine No. (1)	mg/g	433	444	439
Surface Area, BET N_2 (1)	m^2/g	516		
*Molasses Decolorizing Index	-	10.2	10.2	10.2
Moisture, w.b.	%	53.9	53.9	53.9
*Volatile Matter, d.b.	%	11.7	11.9	11.8
*Ash, Total	%	14.78	14.74	14.76
Particle Size (U.S. Sieve #)	nominal	10 x 30		
Oversize (10)	%	0.1		
Undersize (30)	%	2.2		
Effective Size (10% smaller than)	mm	0.80		
Uniformity Coefficient (60%/10%)		1.51		
Mean Particle Diameter (Calc'd)	mm	1.18		
Median Particle Diam. (Graph 50%)	mm	1.18		
*Apparent Density	g/ml	0.424	0.424	0.424
*Modified Phenol Value, MPV				
Westvaco method, 790 Const.	ppm	51.9		
AWWA B600-78, 90 Const.	g/l	5.91		

NOTES AND REMARKS:

(1) Micromeritics automated method, Run No. 800317A

*Corrected formoisture remaining in the dried sample (trace)

Signed:

Stanton B. Smith, Ph.D.
Principal Research Scientist

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ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

CARBON ANALYSIS REPORT

April 11, 1980

SAMPLE:

Source: Shirco, Inc. Grade (if known): Darco HD 1030

Designation: Evansville, Ind. "Feed Composite", 2nd Cycle, Jan. 21-22 '80

Project No.: A-2467 Date Rec'd: 2/20/80 and 3/3/80 (Combined)

ANALYTICAL RESULTS (by Westvaco Standard Methods unless otherwise noted)

TEST	Units	RESULTS Replicates (1)	(2)	Average
Abrasion No. (Ro-Tap)	%	48.4		
* Iodine No.	mg/g	432	432	432
Surface Area, BET N_2 (1)	m^2/g	-		- 636 (After calcining)
* Molasses Decolorizing Index	-	9.7	9.8	9.8
Moisture, w.b.	%	51.9	53.2	52.6
* Volatile Matter, d.b.	%	12.6	12.2	12.4
* Ash, Total	%	14.79	14.74	14.77
Particle Size (U.S. Sieve #)	nominal	10 x 30		
Oversize (10)	%	0		
Undersize (30)	%	5.4		
Effective Size (10% smaller than)	mm	0.70		
Uniformity Coefficient (60%/10%)		1.67		
Mean Particle Diameter (Calc'd)	mm	1.11		
Median Particle Diam. (Graph 50%)	mm	1.09		
* Apparent Density	g/ml	0.434	0.434	0.434 (0.448 after calcining)
* Modified Phenol Value, MPV				
Westvaco method, 790 Const.	ppm	37.2		
AWWA B600-78, 90 Const.	g/l	4.24		

NOTES AND REMARKS:

(1) Micromeritics automated method, Run No. 800 317B

* Corrected for moisture remaining in dried sample (trace)

Signed:

Stanton B. Smith, Ph.D.
Principal Research Scientist

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ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

CARBON ANALYSIS REPORT

April 11, 1980

SAMPLE:

Source: Shirco, Inc. Grade (if known): Darco HD1030

Designation: Evansville, Ind. Regenerated Composite, 2nd Cycle, Jan. 21-22 '80

Project No.: A-2467 Date Rec'd: 2/20/80 and 3/3/80 (Combined)

ANALYTICAL RESULTS (by Westvaco Standard Methods unless otherwise noted)

TEST	Units	RESULTS Replicates (1)	(2)	Average
Abrasion No. (Ro-Tap)	%	50.7		
*Iodine No. (1)	mg/g	595	596	596
Surface Area, BET N_2 (1)	m^2/g	644		
* Molasses Decolorizing Index	-	14.6	14.7	14.7
Moisture, w.b.	%	57.1	56.9	57.0
* Volatile Matter, d.b.	%	6.7	6.4	6.6
* Ash, Total	%	15.65	15.68	15.67
Particle Size (U.S. Sieve #)	nominal	10 x 30		
Oversize (10)	%	0.1		
Undersize (30)	%	4.1		
Effective Size (10% smaller than)	mm	0.74		
Uniformity Coefficient (60%/10%)		1.59		
Mean Particle Diameter (Calc'd)	mm	1.12		
Median Particle Diam. (Graph 50%)	mm	1.10		
* Apparent Density	g/ml	0.402	0.404	0.403
* Modified Phenol Value, MPV				
Westvaco method, 790 Const.	ppm	28.5	28.4	28.5
AWWA B600-78, 90 Const.	g/l	3.25	3.24	3.25

NOTES AND REMARKS:

(1) Micromeritics automated method, Run No. 800 317C

* Corrected for moisture remaining in the dried sample (trace)

Signed

Stanton B. Smith, Ph.D.
Principal Research Scientist

An Equal Employment/Education Opportunity Institution

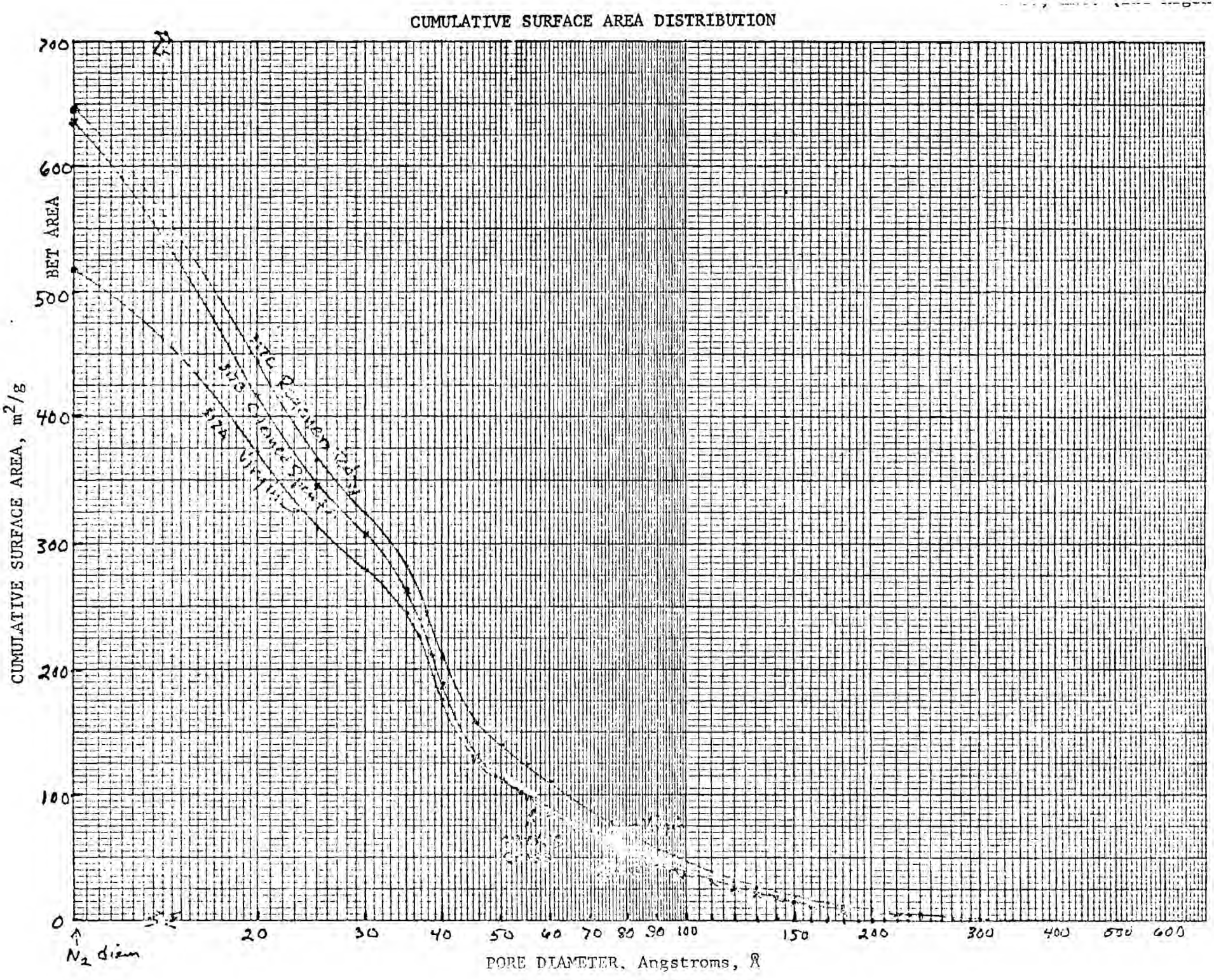

CUMULATIVE SURFACE AREA DISTRIBUTION
CUMULATIVE SURFACE AREA, m²/g
BET AREA
700
600
500
400
300
200
100
0
20
30
40
50
60
70
80
90
100
150
200
300
400
500
600
N₂ diam
PORE DIAMETER, Angstroms, Å

FIGURE 2

Particle Size Distribution

SHIRCO, INC. (2nd Regen.)

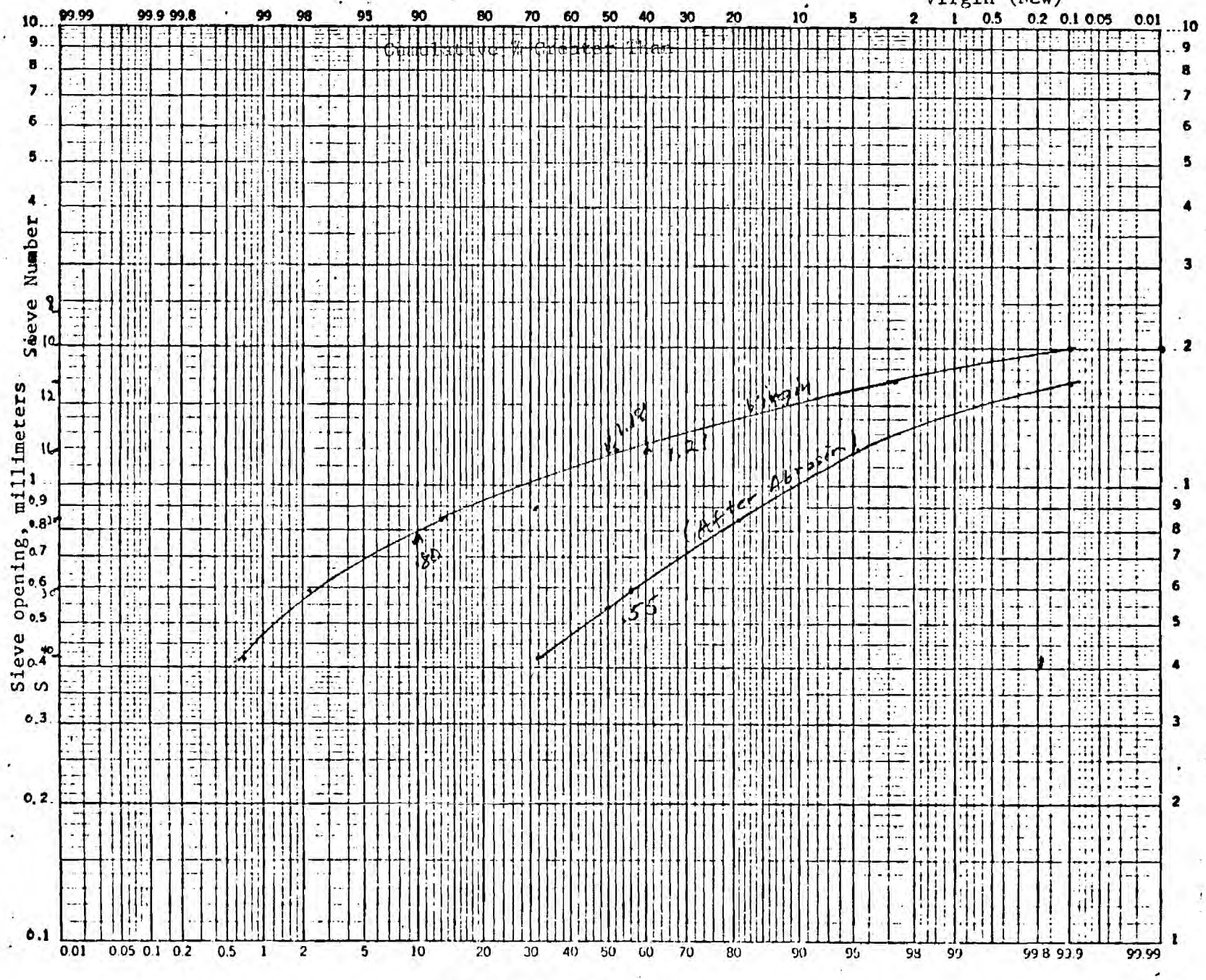


FIGURE 3

PARTICLE SIZE DISTRIBUTION

SHIRCO, INC, (2nd Regen.)
Feed Composite

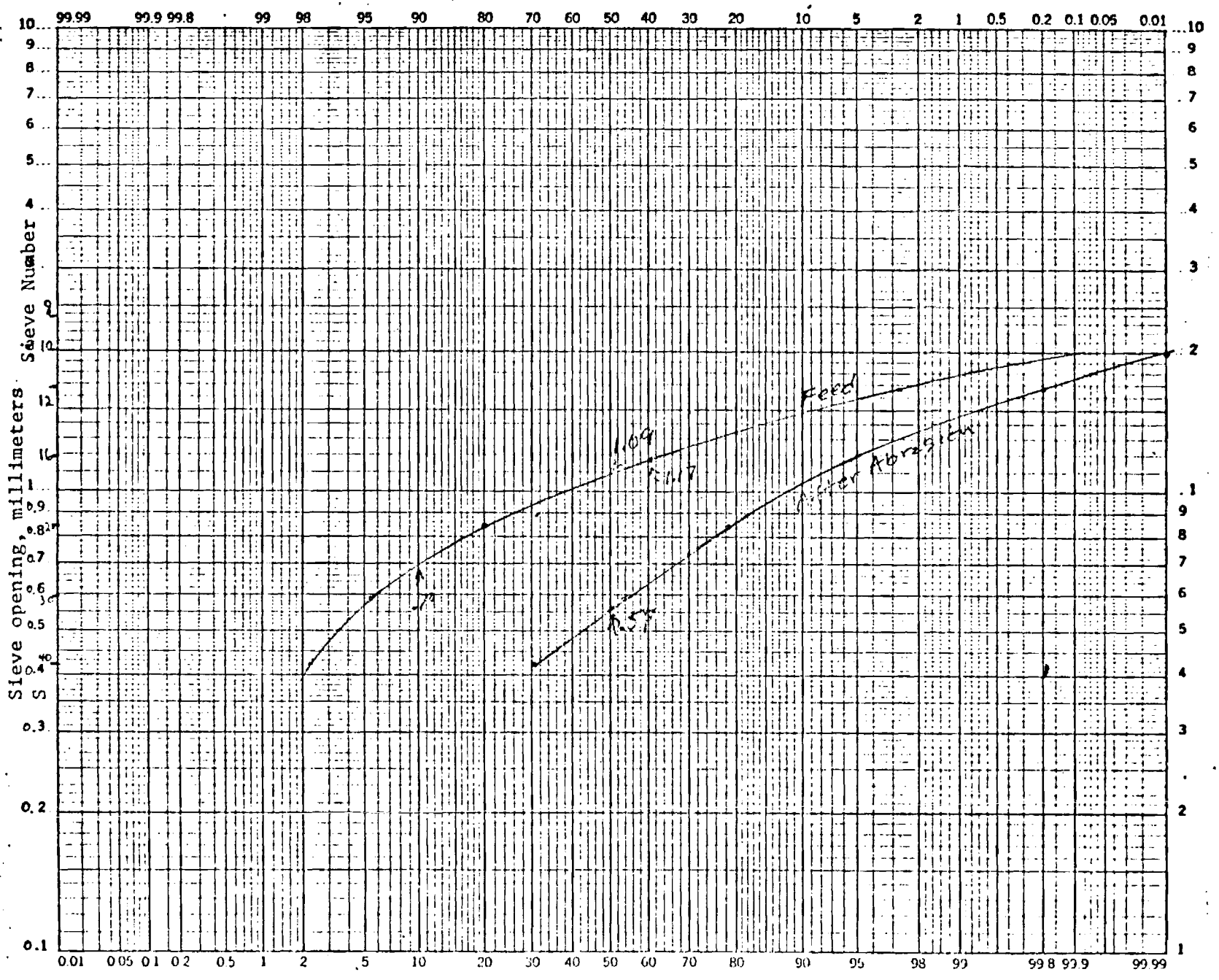


FIGURE 4

PARTICLE SIZE DISTRIBUTION

SHIRCO, INC. (2nd Regen.)

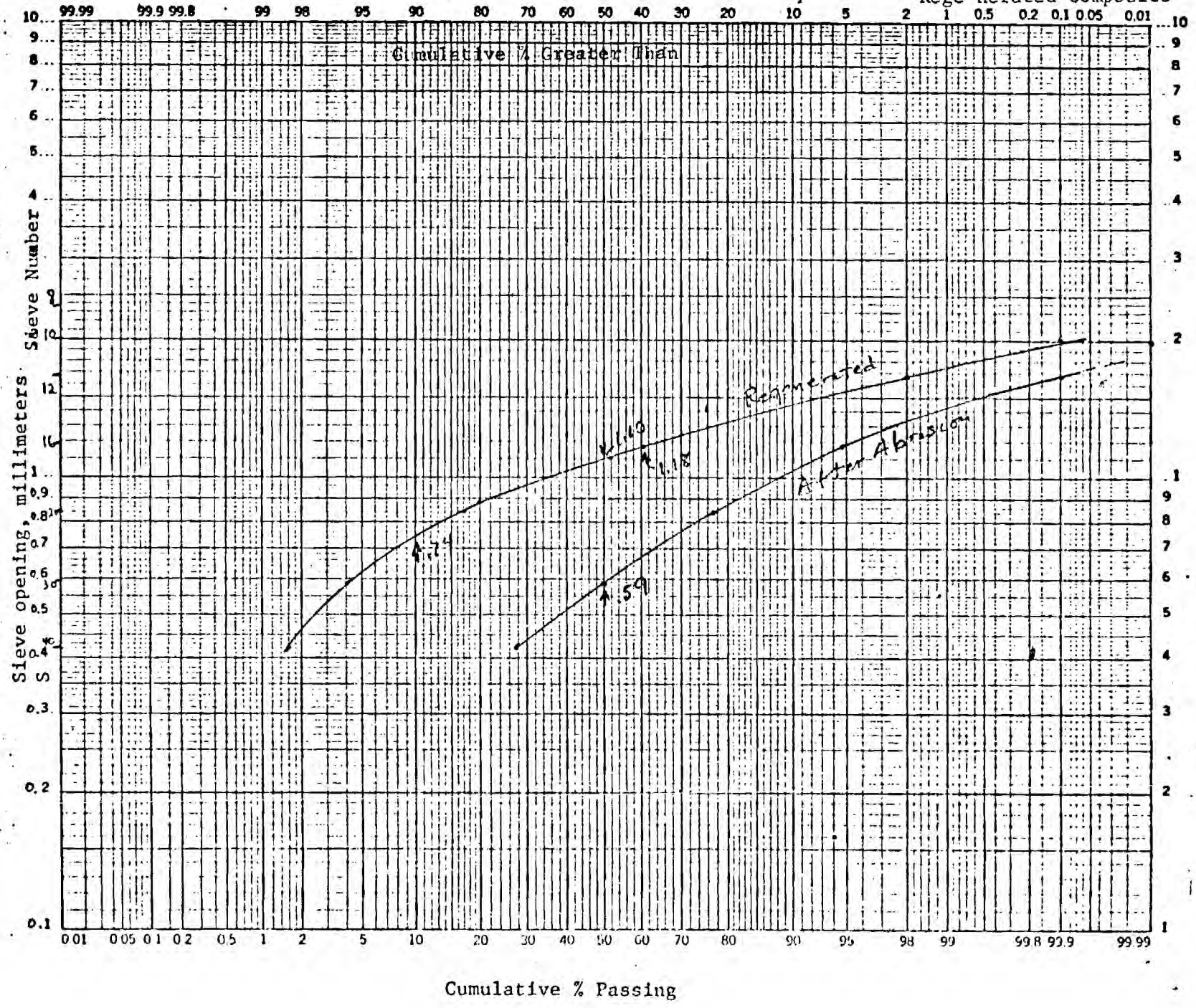
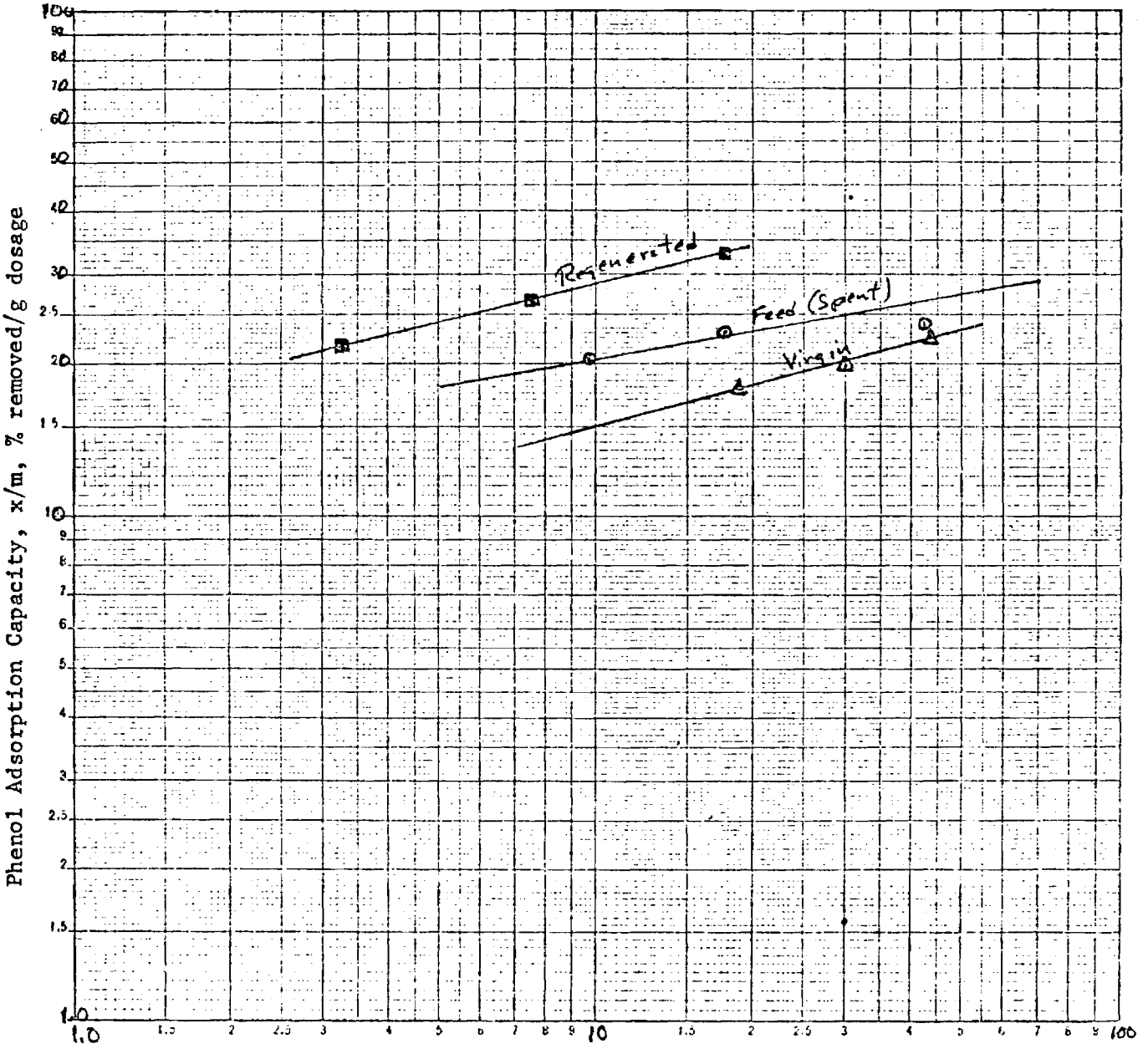


FIGURE 5

PHENOL ISOTHERMS SHIRCO, INC. (2nd Regen.)

for MPV Determinations



ENGINEERING EXPERIMENT STATION
GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

May 20, 1980

Project No. A-2364
Letter Report

Mr. Donald Tiggelbeck
Tigg Corporation
Box 11661
Pittsburgh, PA. 15228

Dear Don:

Here are the rather disappointing results of our study on the gas phase possibilities of activated coal char. Unfortunately, we were led into false optimism by the high saturation capacities for carbontet in the first few tests without realizing that the dynamic flow CCl_4 tests would yield only about half these values.

The large particle work was started in early February using the full range 3/8" x 10 mesh sample. The screen analysis is shown in Figure 1 and Table 1. The +10 mesh is a little high, 12.7%, for 4 x 10 but after activation it would probably be near 5%. Two 1700° F, 100% steam activations at 30 and 40 minutes resp. gave CCl_4 capacities of 51.1% and 67.5% when determined by tne static saturation method overnight. The data is shown in Table 2. However, when the weighing bottles were left in the desiccator containing CCl_4 over the weekend we got much higher capacities. We were therefore, suspicious that condensation was taking place because of temperature fluctuation in the lab. Several attempts were made to determine reliable saturation values using an oven held slightly above room temperature. However, CCl_4 capacities at different lengths of time were all over the map and we finally gave up and decided we should work only with the flow (or dynamic) system where the vapors are not saturated but rather partially saturated at 25°C (air saturated at 0°C but reheated to 25°C). Fortunately, it took the technician only a few hours and some instruction on my part to get the apparatus running reliably.

Two more activations were made using only the 6 x 8 fraction so that a CWS hardness could be run on the product. A slightly lower temperature was used, Ca 1675°F for 30 and 35 minutes resp. However, the dynamic CCl_4 capacities were very disappointing at only half the desired values, even at 35 minutes and less than 50% yield. Two more activations were run on 4/30 at 40 minutes each at two temperatures , 1690 and 1755°F, but the high temperature run was much poorer as shown by the square points (⊡) in Figure 2. One more activation was made using a low temperature 1575° for 60 minutes before giving up. Figure 2 shows that up to 1700°F the temperature made little difference and all the 6 x 8 points fall into line except for the 1755°F run. Also, the "whole sample" run shown by the triangular points (▲) is almost in line as well.

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Visual inspection of all these products was disappointing as they were black and dusty with evidence of severe softening and breakage. It did not seem worthwhile to go on with the extra costs of determining hardness and/or abrasion numbers.

By way of explanation, it appears that the coal char has a strong tendency to develop large pores even at low temperature. Thus, it is well-adapted to making a decolorizing carbon but not a gas phase type. It lacks the versatility of Calgon or Westvaco compacted-and-baked coal which gives good gas phase products at low temperatures but can produce decolorizing carbon at high temperatures with air along with the steam. I suppose the fact that there is a big difference between dynamic and saturation carbontet's might indicate a good "working capacity" for butane adsorption suitable for automotive emission control canisters. However, because of the softness of the product I doubt that this carbon would be suitable in any case. I believe this coal char in the fine sizes is a very promising material and has potential as a decolorizing carbon competing with Darco granular, against which it has a hardness advantage. It also may be useful as a make-up carbon for Calgon F400 and Westvaco WVG. So far the regeneration tests on 2:1 mixtures appear promising.

Very truly yours,

Stanton B. Smith, Ph.D.
Principal Research Scientist

SBS:gjp

xc; J.L.Carden
H.O.Spauchus
OCA

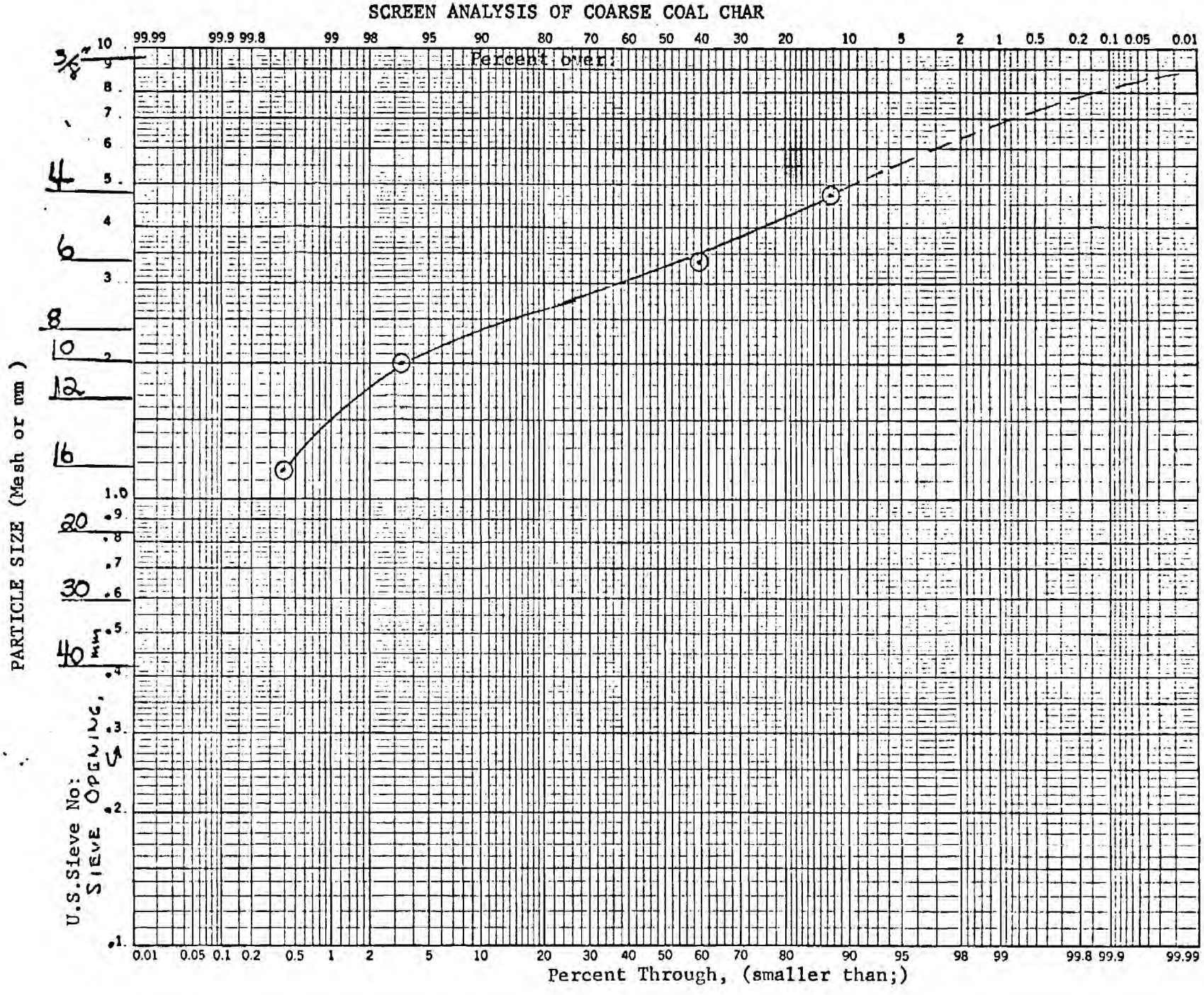
SCREEN ANALYSIS OF COARSE COAL CHAR
Percent over:
99.99 99.9 99.8 99 98 95 90 80 70 60 50 40 30 20 10 5 2 1 0.5 0.2 0.1 0.05 0.01
PARTICLE SIZE (Mesh or mm)
U.S. Sieve No: Sieve Opening, in
3/8" 4 6 8 10 12 16 20 30 40
10 9 8 7 6 5 4 3 2 1.0 .9 .8 .7 .6 .5 .4 .3 .2 .1
0.01 0.05 0.1 0.2 0.5 1 2 5 10 20 30 40 50 60 70 80 90 95 98 99 99.8 99.9 99.99
Percent Through, (smaller than;)

FIGURE 2

CCl_4 CAPACITY OF COAL CHAR vs YIELD & A.D.

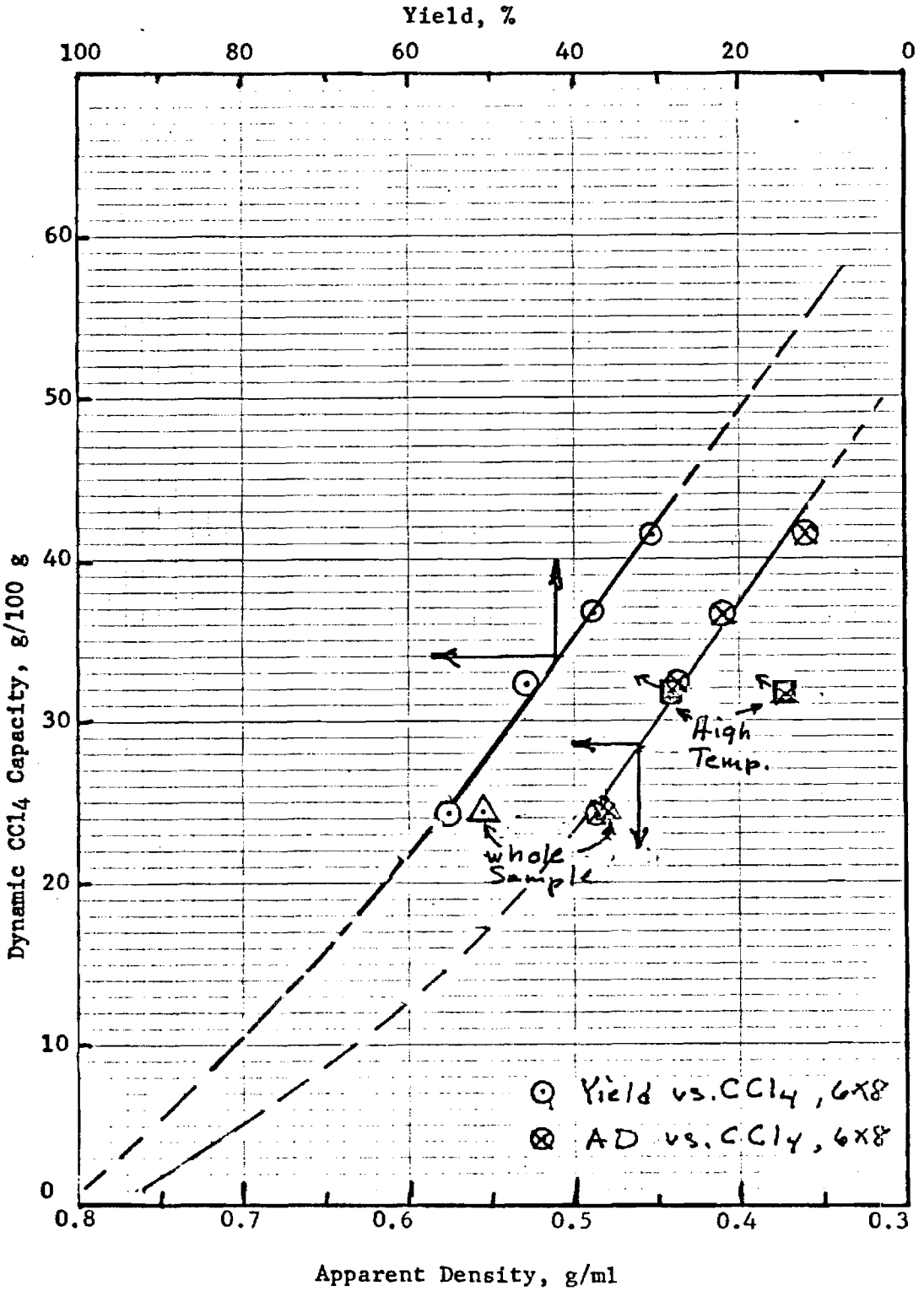

TABLE 1

SCREEN ANALYSIS OF COAL CHAR

U.S.Sieve No. or Opening (in)	Wt. Retained %	Amt Passing Cum. %
3/8"	0	100
4	12.9	87.1
6	27.8	59.3
10	56.0	3.3
16	2.9	0.4
Pan	0.4	

Tigg Corp. Project No. A-2364

TABLE 2

ACTIVATION OF COAL CHAR FOR DEVELOPMENT OF CARBON TETRACHLORIDE CAPACITY

Activation Date	Coal Char Fraction	Activation Conditions			Yield	App. Density	Iodine Number	Carbon Tet Capacity		Remarks
		H_2O g/min	Temp. °F	Time Min.	%	g/ml	mg/g	Static %	Dynamic %	
11/8/79	Fine Part. Sample (Comp.)	2	1570*	33	55.4*	0.502	828	65.7	-	Abr. No. 75.3%
2/11/80 #1	Large Part. Sample 3/8"x10"	2	1700	30	51.2	0.48	666	55.8 (65.9)	24.4	( ) Over weekend
2/11/80 #2	Large Part. Sample 3/8"x10"	2	1700	40	37.0	0.43	713	71.5 (82.4)	-	( ) Over weekend
-----	Raw Material (Reference)		--		(100)	0.76	300	2.9 (9.6)	-	( ) Over weekend
4/17/80 #1	Screened 6 x 8	2	1663	30	55.2	0.484	nd	nd	24.2	
4/17/80 #2	Screened 6 x 8	2	1690*	35	45.9*	0.436	nd	nd	32.2	
4/30/80 #1	Screened 6 x 8	2	1755	40	28.2	0.37	nd	nd	31.7	
4/30/80 #2	Screened 6 x 8	2	1690	40	37.8	0.41	nd	nd	36.7	
5/1/80	Screened (6 x 8)	2	1575	60	30.4	0.35	nd	nd	41.6	
Reference WVH (Westvaco) (dried only)		-						nd	83.0	Unusually high activity sample

*Average values of several runs used to give composite sample

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Phone: 404/424-9651

June 4, 1980

Mr. Donald Tiggelbeck, Pres.
Tigg Corporation
Box 1661
Pittsburgh, Penn. 15228

Re: Letter Report: Project No. A2364
Regeneration Tests

Dear Don:

This letter will attempt to explain in more detail the data sent you in the rough draft of Table 1 included with my letter of May 20th. In the interest of keeping the cost low we followed your instructions to the letter and have not run extra regeneration runs to obtain enough material for hardness tests. We felt that this could be done later if you agreed that the results were interesting enough to warrant it.

We first determined the iodine numbers and volatile matter on each of the samples, the two from Ciba-Geigy and the one from Cargill. We also rechecked a sample of Calgon F400; it was necessary due to a miscalculation of the amount of carbon required so that one of the Ciba-Geigy's and a Cargill sample had to be rechecked in order to put them within the limits for extrapolation of the corrected iodine number. The apparent densities were also determined prior to running activations in the two-inch furnace.

We picked conditions which were fairly close to those found to efficiently activate the coal char in earlier tests which were reported to you in our letter of January 8th. That is, activation with steam at 1575°F. However, since in a real situation regeneration is usually done with a small excess of air on the burners, we calculated the amount of air required to reach a 2% oxygen concentration in the presence of the two grams per minute of water vapor. This turns out to be 300ml of air per minute. On the Cargill sample activation times of 20 minutes and 25 minutes were run. After the 20 minute sample gave us an apparent density of .527 it seemed logical to increase the time slightly and the extra five minutes brought the density to .504.

On the Ciba-Geigy spent sample we started with 20 minutes but in this case got a density of .469 which is somewhat lower than the Calgon F400. Therefore we backed away on the second run to only 17 minutes and this gave us a density of .497 which we felt was close enough to the .50 target value. It was interesting to note that on the Ciba-Geigy sample that even the 17 minute run reached an iodine number of 1043 which is considerably higher than their regenerated sample

or the Calgon F400. In the case of the Cargill sample even the 25 minute run brought the iodine number only to 833 but no longer runs were attempted since we were quite close to the goal on apparent density.

We then made up mixtures of the spent carbons and the fine coal char to give a 2:1 ratio on a volume basis, that is, 2 parts spent carbon, 1 part coal char. This was done by calculating the weights required from the apparent densities. 50 gram portions of these mixtures were then activated as follows.

The Ciba-Geigy mixture at 17 minutes indicated that the iodine number was still low and the apparent density rather high so an additional run at 25 minutes was carried out which still did not quite reach the goal of .500 apparent density but nevertheless gave an iodine number which was close to the Ciba-Geigy regenerated sample and was equivalent to it at least on a volume basis.

For the Cargill mixture, activation times of 25 and 33 minutes were used but it appears we are still fairly far from reaching the apparent density goal since the 33 minute value is still .522. The iodine number was only 725 but it was decided not to do further runs until checking with you on our results and after evaluating the decolorizing index values on all the samples.

In order to save time and money we waited until the end before running the DIs so that we could do them all in one day and we were relatively happy with the results. One sample, the 17 minute regenerated Ciba-Geigy mixture seemed rather high in comparison with the 25 minute value so it was rechecked and found to be about 7.7. If the Ciba-Geigy regenerated sample is used as an index we appear to have accomplished the objective in the 25 minute sample since a DI of 8.5 was obtained. This, however, is not as high as the 20 minute lab regenerated sample which by itself gave 14.4. (This, by the way, was reported in error as 4.4 on the table we sent.)

In the case of the Cargill material the 33 minute regeneration reached a DI of 9.8 which is very close to the 25 minute value on the material by itself. This is perhaps as far as you would want to go in a single pass through the furnace in order that the hardness might be maximized. The value does seem low, however, in consideration of our earlier work where a 33 minute run with steam alone at about this temperature gave a DI of 13.7. This is probably due to the fact that the presence of air is not helping the yield/activity reglationship. It might be that steam alone would be more efficient on regeneration of DI on this particular material. However, since most plant scale regeneration furnaces are directly fired, one would expect to have considerable CO_2 and probably some free oxygen in the atmosphere. It might be well to try simply steam and CO_2 with the mixed carbons and see if this is any more efficient.

After you have had time to study these results lets discuss whether any further work is desired either in more accurately pinning down the most desirable time and atmosphere relationship or whether additional runs might be made to

give us enough for an abrasion number determination. Visually, the samples look quite good and I don't believe there has been an undue breakdown of the coal char particles in these short activation periods.

Very truly yours,

Stanton B. Smith
Principal Research Scientist

SBS:pr

bcc: Hans Spauschus
File, A2364
✓CA

TABLE 1

REGENERATION STUDY ON SPENT CARBON - COAL CHAR MIXTURES

DATE and RUN	BASE SAMPLE	TREATMENT CONDITIONS				YIELD %	APPARENT DENSITY g/ml	IODINE NO. BY:		DECOLORIZING INDEX BY:		VOLATILE MATTER %	REMARKS
		TEMP °F	TIME min	ATMOSPHERE gH_2O min	ml air min			WT mg/g	VOL mg/ml	WT DI/g	VOL DI/ml		
Ciba-Geigy													
-	Spent	-	-	-	-	-	.693	412	286	nd	-	27.3	Diff 21.7, Calc'd Yield 78.3%
-	C-G Regen'd		-	-	-	-	.513	857	439	8.0	4.1	5.6	
5/16/80 #4	Lab Regen'd	1575	17	2	300	69.8	.497	1043	518	13.2	6.56		
5/16/80 #3	Lab Regen'd	1575	20	2	300	69.0	.469	1066	500	14.4	6.75		(Note corrected DI)
5/20/80 Mix, #7	C-G Sp/CoalC 2/1 (v/v)	1575	17	2	300	78.0	.555	794	441	8.2 (7.7)	4.55		Coal Char needs more time ( ) recheck
Mix, #8	ditto	1575	25	2	300	73.8	.531	829	440	8.5	4.51		Better than C-G regen'd
Cargill													
-	Spent	-	-	-	-	-	.681	249	170	nd	-	27.0	Expected Regen. Yield< 78%
5/16/80 #1	Lab Regen'd	1575	20	2	300	74.4	.527	734	387	8.7	4.59		
#2	Lab Regen'd	1575	25	2	300	72.2	.504	833	420	10.1	5.09		
5/19/80 Mix, #5	Car./CoalCh. 2/1 (v/v)	1575	25	2	300	77.6	.560	662	371	7.6	4.26		Coal C needs more time
Mix, #6-2	ditto	1575	33	2	300	71.0	.522	725	378	9.8	5.12		Not quite there
Reference Samples													
-	F400 Calgon	-	-	-	-	-	.500	930	465	12.6	6.4		(223 Mol. No.)
11/8/79	Coal Char	1575	33	2	0	55.4	.500	828	414	13.4	6.7		Previous Study